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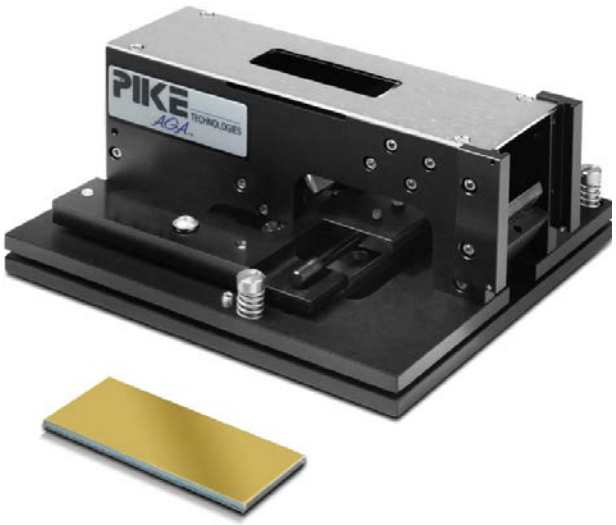
Spectroscopic Creativity

Installation and User Guide



AGA II™ Advanced Grazing Angle Specular Reflection Accessory

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Contents

Introduction	1
Unpacking Your Accessory	2
Packing List	2
Optical Path	3
Alignment	4
Establishing the Sample Beam Location	5
Sample Measurements	5
Use of Polarizers	6
Application	7
Hard Disk Analysis	7
Precautions	9
Mirrors	9
References	9
Replacement Parts and Options	9

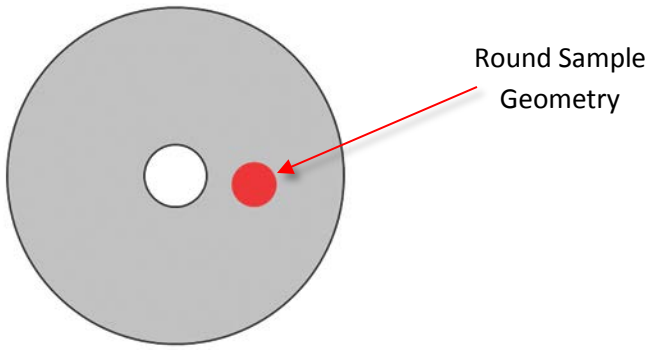
Introduction

Measurement of very thin layers, down to monomolecular coverage, on reflective surfaces is a very demanding application. Thin layers for catalytic studies, coatings on medical devices, biological and chemical sensors all need to verify coating thickness and quality. All of these applications can benefit from grazing angle measurements.

One major application of grazing angle is the manufacture of magnetic disks. Fluorocarbon lubricants are applied to the disk surface. This lubricant layer is used to reduce head wear during operation and to reduce friction during start up and landing of the head. Knowledge of the thickness of the lubricant layer is critical in predicting disk performance. An excess or shortage of lubricant can adversely affect disk drive life. Application of the lubricant to the disk surface is commonly done by spinning. Although this produces a very thin lubricant layer, the thickness of this layer may vary over the surface of the disk. It has been demonstrated that storage of the disks on edge can cause a significant variation in lubricant thickness across the disk, the layer being thin at the top and thick at the bottom[1]. Not only must the overall lubricant thickness be determined, but it is also useful to map variations in the lubricant thickness across the disk.

A common method for the non-destructive measurement of this lubrication layer is by performing an infrared specular reflectance analysis[2]. The analysis is complicated somewhat in that the lubrication layer is placed over the magnetic medium, instead of on the metallic disk surface. The lubrication layer is also very thin, its thickness being much less than the wavelength of the light used for analysis. Greenler[3] has shown that the sensitivity of a measurement of a thin film on a reflecting metallic substrate can be significantly enhanced by performing the analysis at a grazing angle using light that has its E vector perpendicular to the plane of incidence (parallel or p-polarization).

The AGA is unique among other grazing angle reflectance accessories in that there is a discrete selector for the size of the spot measured on the sample surface. The spot is round and only the size selected by the PIN mirrors as shown below.



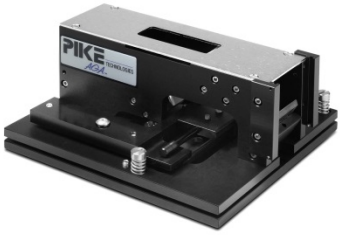
Unpacking Your Accessory

In order for you to quickly verify receipt of your accessory, we have included a packing list. Please inspect the package carefully.

Packing List

AGA Accessory User Manual
PN 350-015000
Quantity 1

AGA Accessory
PN 015-10XX
Quantity 1



Gold Alignment Mirror
PN 300-0002
Quantity 1

Balldriver Hex Wrench Set
Quantity 1

Optical Path

The key element of the advanced grazing angle accessory is the pin mirror assembly. This accessory has been designed to optimize grazing angle measurements. The size and shape of the spot on the sample is well controlled, circular and is selectable, defined by the optics contained in the accessory. The optical design maximizes throughput so that good spectra may be obtained from very small samples. Five mirrors are mounted onto a slide which has a detent for each mirror. The diameters of the mirrors are 1/8", 3/16", 1/4", 3/8" and 1/2". By moving the slide, the desired pin mirror may be moved into the beam path.

The beam from the spectrometer is focused onto the pin mirror. The angle of incidence of the beam onto this mirror is equal to eighty degrees. The beam that is reflected from this mirror is imaged at unit magnification onto the sample, striking the sample at the same eighty degree angle of incidence. Thus the area of the sample that is illuminated is equal to the size of the circular pin mirror.

The reflected beam from the sample passes through a ZnSe prism which redirects the beam to the detector.

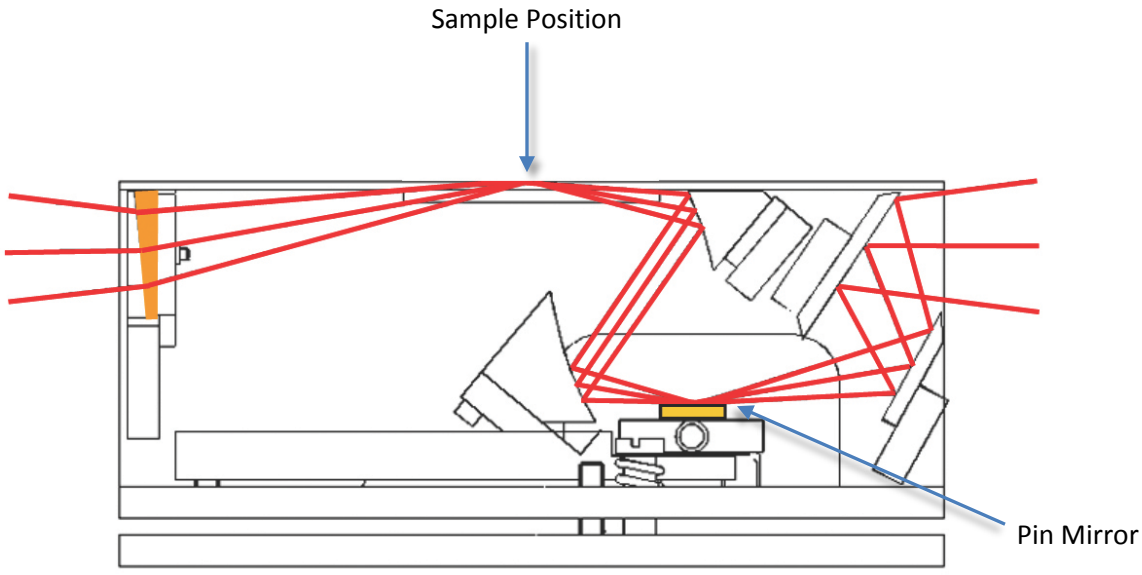


Figure 1. Beam path within the AGA accessory

Alignment

The Advanced Grazing Angle accessory has been pre-aligned. The only alignment necessary is the positioning of the accessory in the FTIR sample compartment and adjustment of the height and tilt using the set screws on the accessory plate. Should you need to align the accessory, please follow the directions below.

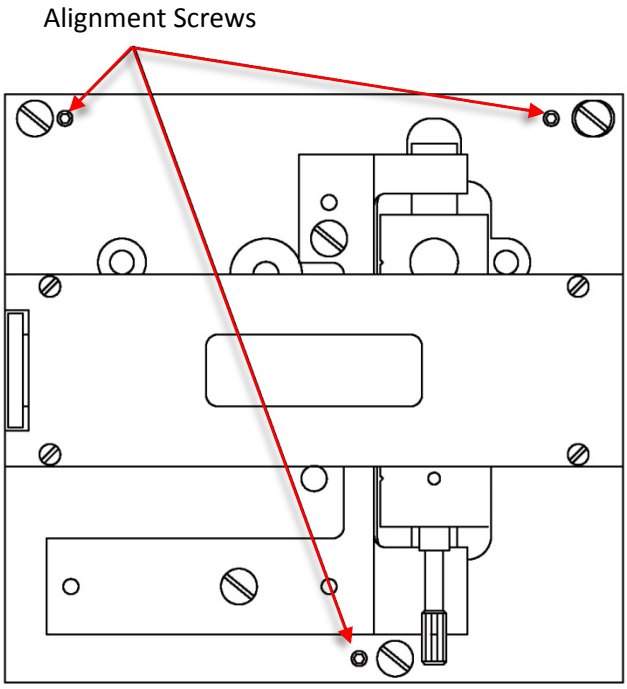


Figure 2. Overhead interior view of the AGA accessory showing location of alignment screws

1. Insert the accessory into the FTIR sample compartment.

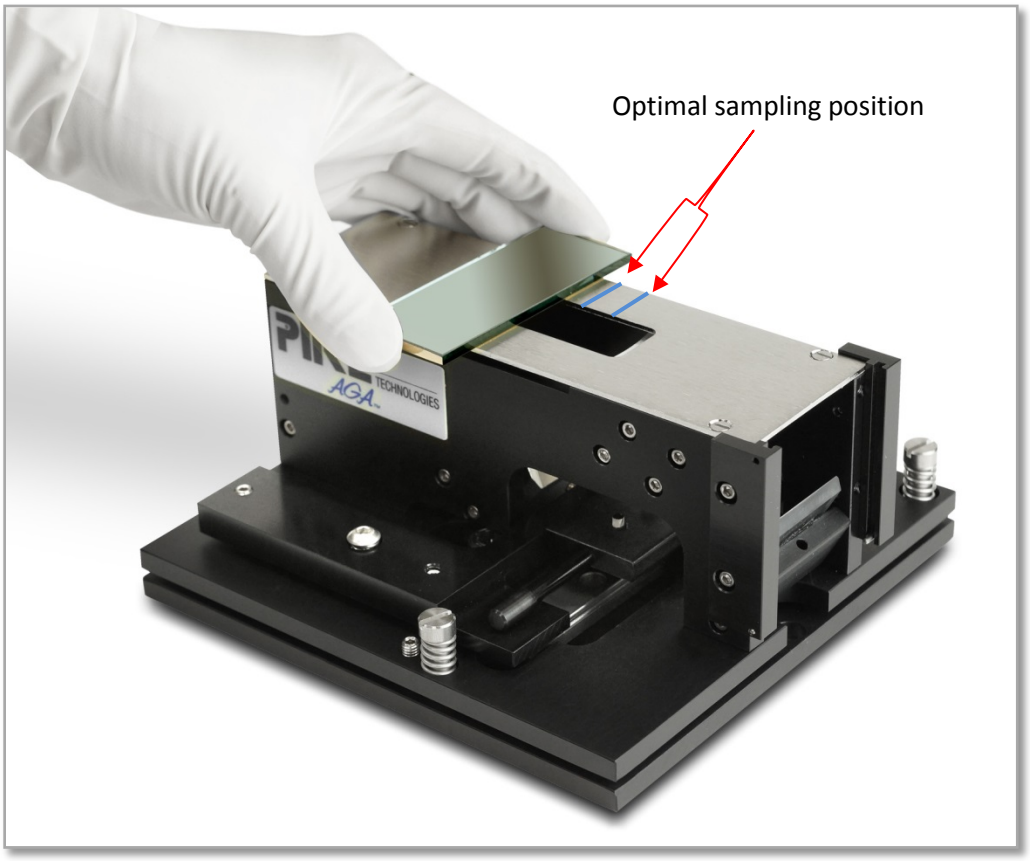
2. Enter the alignment mode or energy throughput mode of the FTIR software.

3. Set the detented slide for the center mirror (1/4”) and place the alignment mirror provided on the top sample surface.

4. While monitoring the size of the signal, adjust the three set screws shown in above for height and tilt until maximum throughput is achieved.

Establishing the Sample Beam Location

With the FTIR in monitor mode, check the energy or the interferogram maximum value. Without any sample it should be close to zero. Using the gold mirror establish the boundaries of the illuminated beam. Slide the mirror from one side until the interferogram values start suddenly increasing (e.g. 5% of the maximum value). Mark the top of the accessory from both directions with a marker. The area within the marks is the illuminated position that must be completely covered by the sample. If the sample is smaller, a smaller PIN mirror needs to be selected.

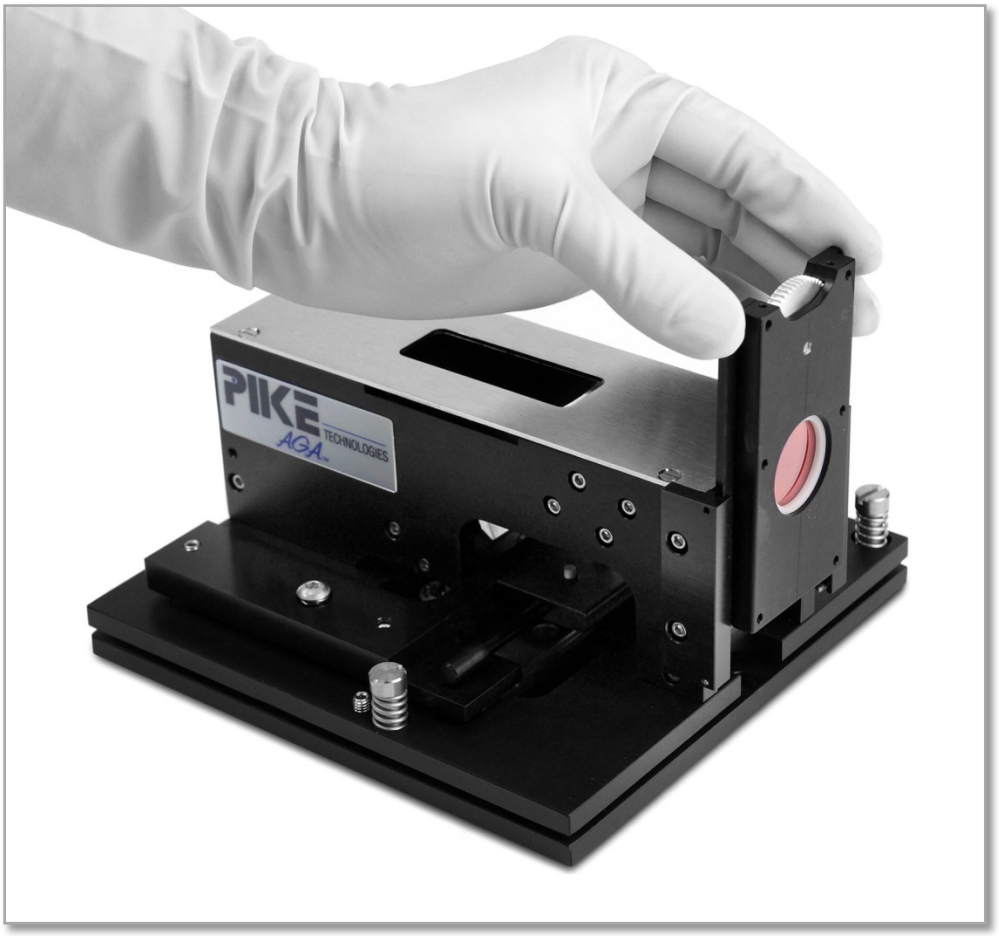


Sample Measurements

Samples are placed on top of the AGA accessory such that the flat sample touches the top of the accessory. It is very important that the repositioning is reproducible, the sample plane is not varying from sample to sample. The grazing angle alignment is very sensitive to vertical positioning of the sample. Please make sure that the angle of the sample is the same for the reference as well as all the samples.

Use of Polarizers

In order to get the best sensitivity, use a polarizer to enhance the signal. A p-polarized light will enhance the surface species measured. Set the dial to zero when using a PIKE polarizer. The polarizer will reduce the overall signal; therefore for optimum results we recommend that a liquid nitrogen cooled MCT detector is used.



Application

A self-assembled thiol monolayer was deposited on a gold mirror to illustrate the power of the grazing angle measurement technique. The polarizer was set to perpendicular to the sample plane. Using an uncoated gold mirror as the background, a very good quality spectrum can be obtained within a one minute measurement time with the MCT detector.

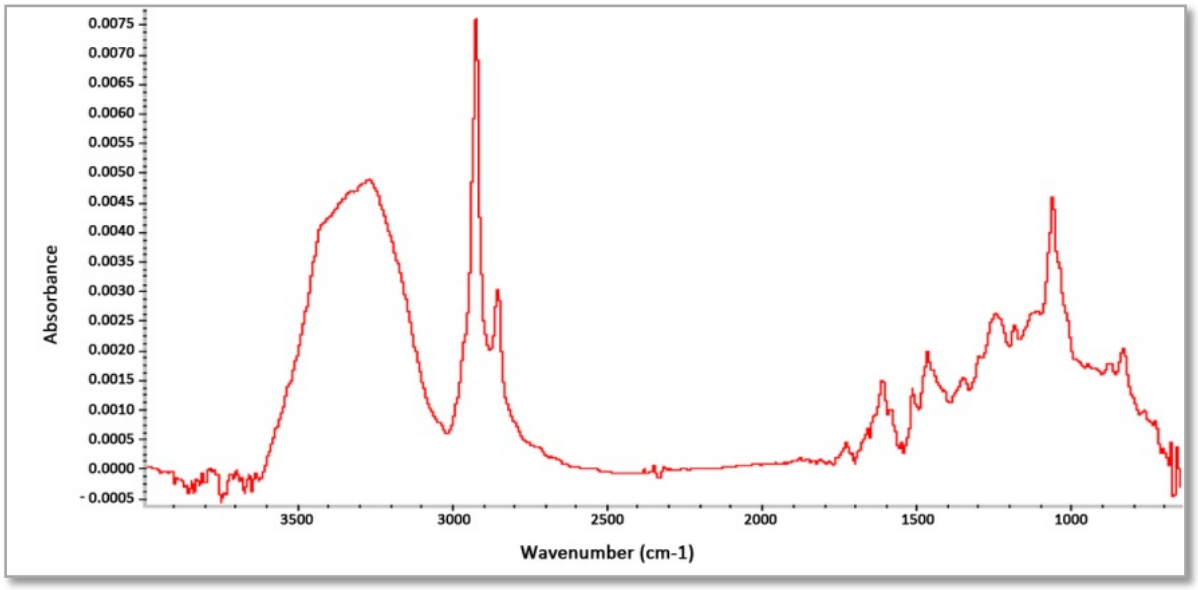


Figure 3. Spectrum of self-assembled thiol monolayer

Please note the scale of the above spectrum. The very thin layers represent a very small absorption, in the order of a few milliabsorbance units. This is why the instrument, the detector, and the purging of the FTIR instrument must be in good condition to obtain good spectral results.

Hard Disk Analysis

For the analysis of large disk platters, such as the popular 5.25 inch disk drives, a useful map of the lubricant thickness over the surface of the platter can be made using a simple eighty degree reflection accessory, even with an elliptical sampling area which is almost two inches long. Over the past few years the size of disk drives has steadily decreased while the capacity of the drives has increased. One of the common sizes of disk platters at the moment is 48 mm diameter. The use of a traditional grazing angle accessory for the analysis of these platters is not acceptable due to the large size of elliptical spot illuminated on the surface of the disk. Good spatial resolution is not possible and at most positions on the disk the spot actually falls off the surface of the disk, resulting in a loss of quantitation.

The Advanced Grazing Angle accessory is capable of measuring even the smallest disks with a good spatial resolution and exceptional signal-to-noise. Films of the order of 20 angstroms thick may be measured to an accuracy of better than one angstrom.

The spectrum shown is of a lubricated disk, ratioed to an unlubricated disk. The lubricant thickness of this sample was 18 angstroms. Sixteen scans were coadded at a resolution of 8 cm^{-1} using an MCT detector. The spot size was 1/8 inch diameter.

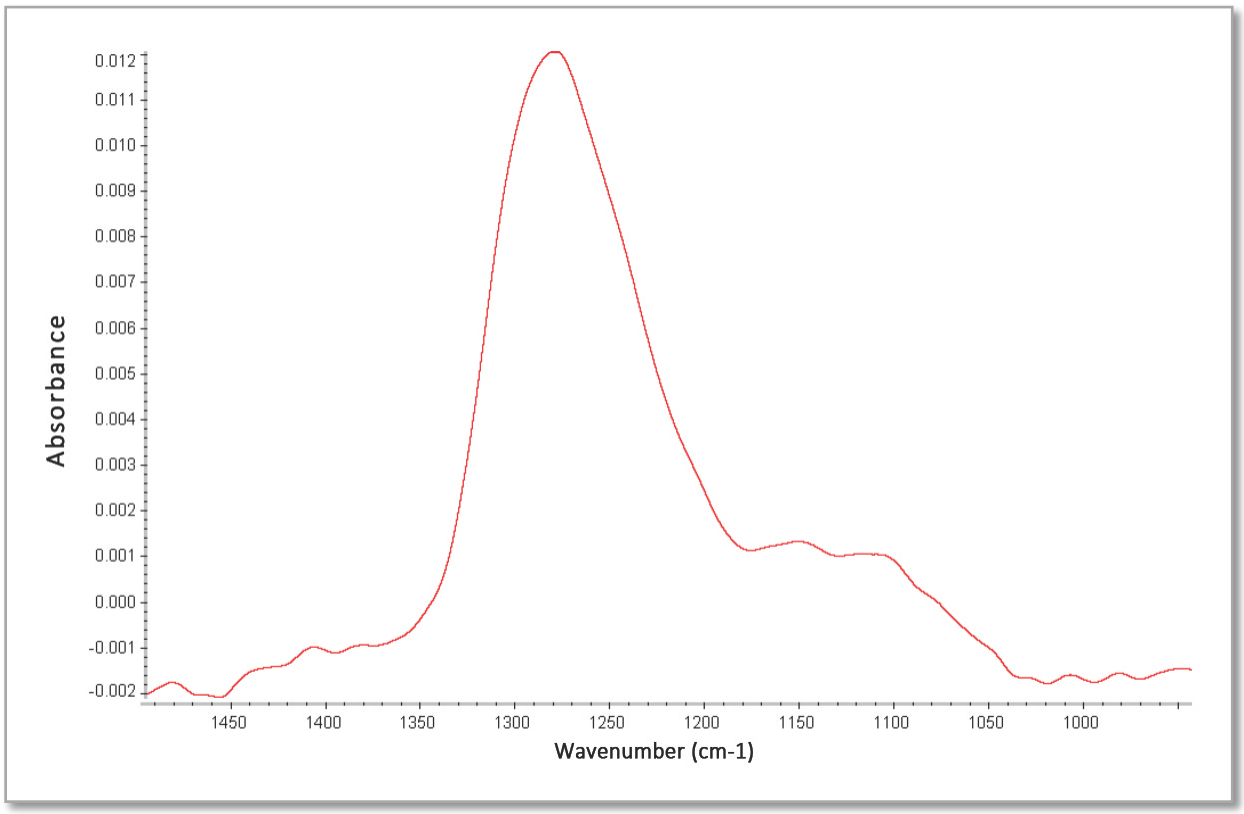


Figure 4. FTIR spectrum of an 18 angstrom thick lubricant on hard disk measured in 15 seconds using an MCT detector.

Precautions

Mirrors

In order to provide the maximum transmission in the infrared, with the minimum spectral interferences, the mirrors used in this device are uncoated (bare) aluminum on glass substrates. Since the coatings are soft, care must be taken to avoid damage. Normally, these mirrors will not need cleaning since they are contained within the housing of the accessory. If they do need cleaning, they may be gently wiped with a lint-free, abrasive-free cloth, such as lens tissue, or with a camel hair brush. Under no circumstances must the mirrors be rubbed with paper products such as “Kleenex” since this will scratch the mirror coating.

References

1. K. Nishikida, Automated Mapping Hard Disk Checker for Lubricant Thickness Determination, Paper No. 779 presented at Pittcon 1992.
2. F. Walder Nicolet Application Note 8313.
3. R. G. Greenler, J. Chem. Phys. 44, 310 (1966).

Replacement Parts and Options

PART NUMBER	DESCRIPTION
300-0002	Gold Substrate Alignment Mirror (1.25” x 3.0”)
090-1000	Manual Polarizer, ZnSe
0901200	Manual Polarizer, KRS-5

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